IMPERIAL COLLEGE OF SCIENCE, TECHNOLOGY AND MEDICINE

Faculty of Engineering

Department of Chemical Engineering

CERAMIC HOLLOW FIBRE CATALYTIC CONVERTERS FOR AUTOMOTIVE EMISSIONS CONTROL

NUR IZWANNE BINTI MAHYON

A Thesis Submitted for the Degree of Doctor of Philosophy and the Diploma of Imperial College London

DECLARATION OF ORIGINALITY

I hereby declare that this thesis and the work reported herein was composed by and originated entirely from me. Information derived from the published and unpublished work of others has been cited, acknowledge in the relevant text and related references are included in this thesis.

Nur Izwanne Binti Mahyon

Imperial College London,

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ABSTRACT

The development of ceramic hollow fibre catalytic converters for the control of automotive emission has been presented in this thesis. Attempts have been made to understand the different factors such as the fabrication of the substrate, the effects of the washcoat packing, the variations of the catalytic reactions at different catalyst formulations, and the evaluation of the pressure drop in the new substrate structure, since these factors may cause a real hindrance in the development of a new ceramic hollow fibre catalytic converter. An asymmetric ceramic hollow fibre substrate was fabricated through the extrusion process, assisted by a phase-inversion. The produced substrate resulted in a hollow fibre with an array of microchannels with almost double the hydraulic diameter of the commercial 400 cells per inch square (CPSI) honeycomb monolith, which lead to less pressure drop in the system. The hollow fibre substrate can offer a tremendous increase in the geometric surface area (GSA), which is beneficial for catalyst layer deposition. With the new structure, a new washcoating technique has been proposed. A loosely packed washcoat in the microchannel has been identified as the best configuration. After the successful conversion of CO at a low light-off temperature and low precious metal loading, two perovskite catalysts have been synthesised, and their catalytic activity in the hollow fibre catalytic converter has been assessed. This result indeed highlights the advantage of the new proposed structure for catalytic converters in order to control tailpipe emissions.

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CHAPTER 1

Introduction

1.1 Background

A report published in 2011 by the World Energy Council (WEC) states that the global transportation sector is expected to face an intensification of unprecedented challenges in the four decades between 2010 to 2050. The Global Transport Scenarios to 2050 has been built to examine the future of this industry that would be profoundly affected by several factors such as global economic growth, demographic trends and any future technological breakthroughs [1]. The proliferation of the transportation industry, driven by population demands, is predicted to cause an increase in global emissions if left unchecked. Further, it is expected that by the year 2050, carbon dioxide (CO_2) emissions will increase by nearly 79%, approximately 12 Gt CO_2eq/year, which is subject to government intervention in a low-carbon transport policy [2]. The aforementioned figure is alarming and has put pressure on policymakers to pursue strict emission regulations within a tighter range of concentration, as a mitigation measure.

Tailpipe emissions originating from an internal combustion engine (ICE) emit a number of combustion by-products. Under normal engine operating conditions, the following is observed as the typical composition of the gases emitted (See Table 1.1).

Table 1.1 Typical exhaust gas composition at normal engine operating condition for gasoline [3]

Major Constituents	Composition
Water, H_2O	10 vol.%
Carbon dioxide, CO_2	10 vol.%
Unburned hydrocarbons, HCs	350 vppm
Oxygen, O_2	0.5 vol.%
Carbon Monoxide, CO	0.5 vol.%
Nitrogen oxides, NOx	900 vppm
Hydrogen, H_2	0.17 vol.%

CO, HCs and NOx are the major pollutants from the exhaust. CO and HCs are formed as a result of incomplete combustion, while combustion at a sufficiently high temperature and pressure produces NOx. Exposure to CO causes detrimental health risks when inhaled, since CO restricts the level of oxygen in the blood and causes further suffocation of the organ through the displacement of oxygen. Also, high concentrations of CO may lead to unconsciousness or even death. HCs and NOx, on the other hand, are responsible for environmental hazards since a reaction between the two compounds and sunlight produces ground-level ozone, which is a major component of smog and other secondary pollutants. Photochemical smog causes a series of respiratory diseases and further leads to the irritation of the eyes, reducing visibility [4,5]. Considering the health and environmental impacts caused by these compounds, the development of exhaust treatment technologies is crucial for minimizing the risks posed by automotive ICE emissions.

To address the risks discussed above, tailpipe emission control devices, known as catalytic converters, have been used to treat internal combustion engine products and convert them into innocuous gases such as CO_2, water (H_2O) and nitrogen (N_2). A tailpipe control device was first invented during the 1950s by the French mechanical engineer Eugene Houdry, an expert in catalytic oil refining [6]. Early published studies concerning smog in Los Angeles brought Houdry's attention to these issues, who then focused on trying to reduce the health risks associated with the increasing levels of air pollution caused by the burgeoning automobile and industrial sectors. Widespread use of the catalytic converter only began in the mid-1970s after the U.S. Environmental Protection Agency (EPA) enabled strict regulation, requiring every gasoline-powered vehicle manufactured 1975 onwards to be equipped with a catalytic converter [7]. Further research was expected to improve the initial designs of the catalytic converter. The original design operated as a two-way converter where it targeted the oxidation of CO and hydrocarbons only. The current generation of the catalytic converter is capable of nullifying CO, HCs and NOx simultaneously, and is known as the three-way catalytic converter. Interestingly, catalytic converters do not have an application in a vehicle exhaust system only, but also have broader uses in emission control for electrical generators, mining equipment, locomotives and aeroplanes.

The conventional catalytic converter is a honeycomb structure in a monolithic configuration made of ceramic or metal components coated with metal catalysts from the Platinum Group Metals (PGM), where the constituent elements are usually platinum (Pt), rhodium (Rh), and or palladium (Pd), subsequently encased in a stainless-steel container. The honeycomb monolith design provides a parallel flow channel for contact between the reactant and catalyst, limiting unnecessary back-pressure in the system. The metal active catalyst layer, also known as a catalytically active washcoat, consists of a high surface area material impregnated with a catalyst. There are two different types of catalysts at work; an oxidation catalyst and a reduction catalyst. Although the research on catalytic converters has been extended to study different types of non-PGM catalysts and compatible oxygen storage material washcoats, this research is still in the experimental stage [8–11]. Nonetheless, the main concern with the non-PGM is their susceptibility towards deactivation due to the sulphur originating from diesel fuel especially, further making such metals unfavourable for long-term usage. Hence, highly valuable and expensive PGMs remain the preferred choice of catalyst after significant experimentation with cheaper alternatives has yielded inferior results [12].

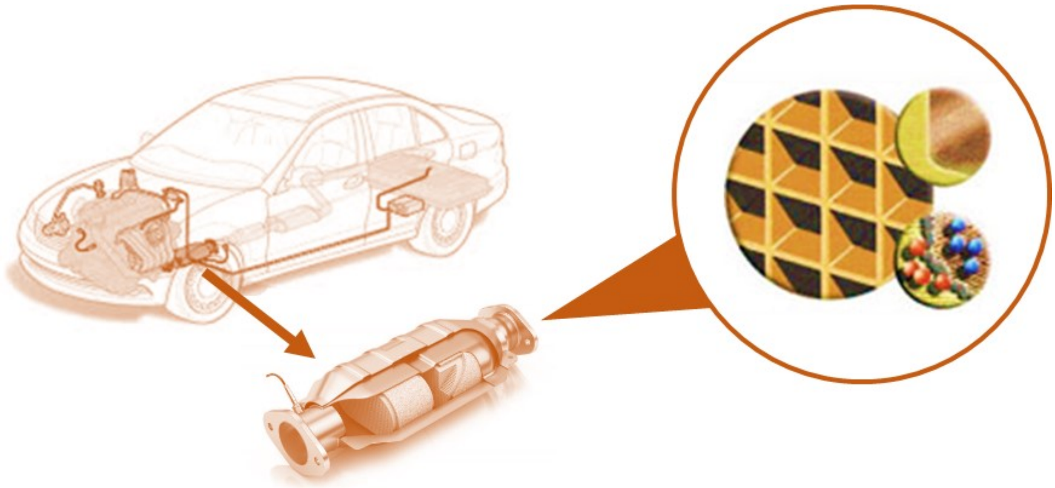

Figure 1.1 Diagram of catalytic converters and its position in the cars

The uncertainty around the future of PGM is largely due to the progressive depletion of the rare metals used. Further, the price volatility of PGM would restricts commercial use of this material for long term [13]. One solution is to reduce the amount of PGM in catalytic converters without affecting their efficiency and performance. As catalytic performance is directly proportional to the contact of the active sites with the reactants, a larger surface area for deposition is required in order to optimise this interaction. This can be achieved by increasing the geometric surface area (GSA) of the substrate on which the catalytic washcoat can be deposited evenly. A common practice to improve the GSA is by increasing the number of cells per inch square (CPSI) of the monolith [14]. However, this method draws a much higher pressure-drop because of increased flow restriction at the catalyst entrance. Therefore, it is crucial that any optimisation of the substrate takes into account two conflicting requirements: (1) the increase of contact area between the gas and the catalyst and, (2) minimisation of pressure loss across the gas flow path [15].

In this thesis, a novel ceramic hollow fibre was fabricated using a single-step phase-inversion extrusion technique. A hollow fibre containing microchannels was used as a new substrate for the application of catalytic converters in pursuance of controlling automotive emissions. The availability of microchannels in the substrate has been proven to offer high GSA without having to restrict the open area entrance for the fluid to pass through, thus, improving the back-pressure condition of the engine. With a significant GSA at hand, the deposition of the active material was done at a notably lower amount than the conventional formulation, reducing the PGM loadings in the new system. The study also explored the most favourable packing conditions of the catalyst in the substrate to fully utilise the active sites and to minimise the mass transfer resistance during the operation. Catalytic performances were carried out to evaluate the conversion efficiency by using CO for a sample reaction.

After the success of the ceramic hollow fibre catalytic converter using a palladium-only catalyst, an attempt to further reduce PGM loading in the catalytic converter led the research to synthesise a palladium-doped perovskite. Finally, two types of palladium-doped perovskites were synthesised in-house, and their conversion performance was studied and discussed.

1.2 Thesis Objectives

The primary objective of this thesis is to develop ceramic hollow fibres for catalytic converters containing a substantially reduced volume of Platinum Group Metals (PGM), for emission control from light-duty vehicles. In this study, ceramic hollow fibres containing microchannels with a high geometric surface area are used as a substrate for catalytic converters. These were fabricated through a phase inversion assisted single-step extrusion process. In order to achieve the primary objective of the study, the following milestones were set to ensure the completion of the project:

i. ***To fabricate defect-free ceramic hollow fibre substrates using a single-step extrusion process***

Porous ceramic hollow fibre substrates were fabricated by the extrusion phase inversion process, using a single orifice spinneret. This step was aimed towards producing a hollow fibre with high porosity and open micropores in the inner surface with a relatively high mechanical strength, for use as a substrate incorporated with the active metal catalyst.

ii. ***To study the effects of the washcoat loadings and the packing of the hollow fibre microchannels on the catalytic reaction of CO oxidation.***

Catalytic performance is not only affected by the reactivity of active metals being used. In catalytic converter applications, the washcoat layer also plays a significant role. The thickness of this layer affects the conversion performance resulting from the existence of the mass transfer resistance in the system. Different washcoat loadings and packing conditions lead to a difference in the mass transfer regime during the reaction. Thus, finding the best condition for the washcoat packing is vital towards ensuring optimal catalyst utilisation.

iii. ***To determine a washcoat and catalyst deposition technique for an even distribution of the active sites onto microchannels.***

Catalytic converters are made of monolith support deposits with washcoat and catalysts. As the new proposed design contained open microchannels, deposition and impregnation of active layers required modifications from conventional practice. Different deposition and impregnation techniques were investigated to achieve well-dispersed and highly distributed active catalyst sites.

iv. ***To explore the options of available low Platinum Group Metals as a potential three-way catalyst.***

Price volatility and the scarcity of PGM makes finding a suitable substitute for this type of catalyst compelling. A Perovskite oxide has proven to have interesting properties. One of the properties of a perovskite oxide is the enhancement of the thermal stability of the easily sintered PGMs while maintaining their reactivity. For this reason, two types of perovskite oxides containing different metals were synthesised. The catalytic activity of precious catalysts and perovskite catalysts was studied by measuring the oxidation reaction of carbon monoxide.

1.3 Thesis Structure and Organisation

This thesis is composed of seven chapters discussing the process and steps taken to use ceramic hollow fibres as the new substrate for catalytic converter applications. The process starts with the fabrication of alumina ceramic hollow fibres as the new substrate design for the catalytic converters. The effects of the washcoat packing inside the microchannels on the CO oxidation performance are studied, followed by the performance of low PGM perovskite oxides catalyst in the hollow fibre substrate. Figure 1.2 presents the overall flow of the thesis.

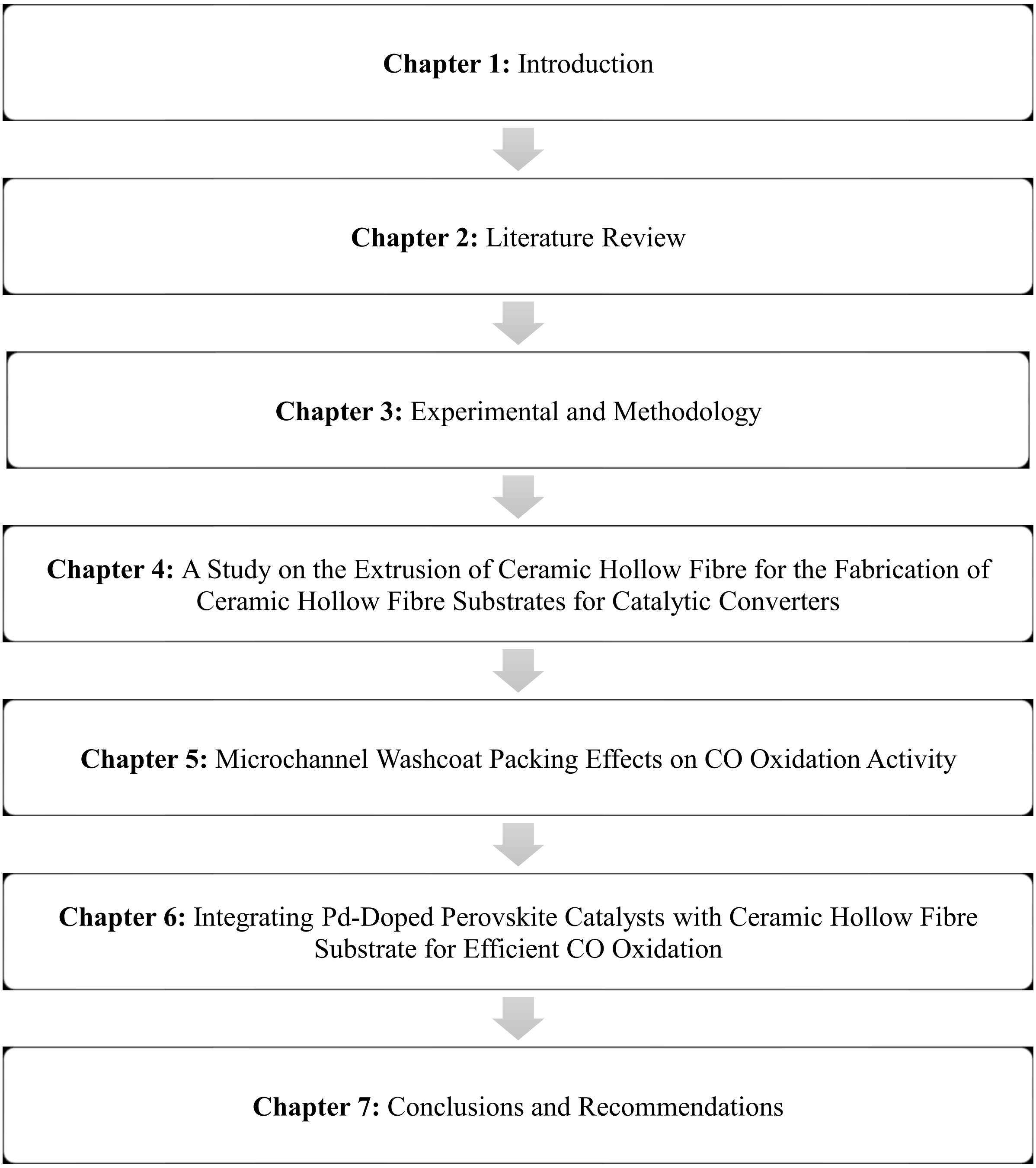

Figure 1.2 Overall structure of the thesis

A brief history and introduction of catalytic converters is summarised in **Chapter 1**. This chapter includes the discussions on the objectives of the study and an overview of the thesis. Subsequently, **Chapter 2** presents the literature review, which provides a more comprehensive discussion on the topic concerning catalytic converter components as well as the existing and the current development of the topic. Catalyst studies and challenges that have yet to be overcome in tailpipe emission treatments are also examined in the review of the literature. Further, ceramic hollow fibre fabrication through phase inversion technique is discussed. The discussion also extends to the application of an emerging perovskite oxide as a new potential catalyst, to substitute the commercially used PGM catalyst.

Chapter 3 lists all materials used in the process, methodology, characterisations and experimental procedures applied to achieve the aforementioned objectives of the study.

Chapter 4 presents the process and success rate of fabricating new ceramic hollow fibre substrates for catalytic converters through the extrusion technique assisted by phase inversion. The formation of the microchannel, through this technique, the morphological evaluation and the structural improvement, as compared to the typical honeycomb structure, is also discussed in this chapter.

Chapter 5 discusses the relevant processes after the success of the substrate fabrication. The chapter discusses the effects of the washcoat packing inside the microchannel. Since a ceramic hollow fibre is new in the use of a catalytic converter application, the configuration and an effective washcoat layer is critical to the study. In addition, CO oxidation reactions were carried out, and their performance was evaluated.

Chapter 6 maintains continuity with the studies concerning the washcoat effect in Chapter 5, examining the objective of reducing the content of PGM applied to the catalytic converter system. For this process, palladium doped perovskites were synthesised, and their morphology, structural and reactivity were characterised. Their reactivity was evaluated by CO oxidation also as a sample reaction, and the effects of the packing configuration in the hollow fibre substrate were compared with the packed-bed packing. Mass transfer limitations in the washcoated hollow fibre substrates are further discussed in the chapter.

Finally, all findings from the study are summarised in **Chapter 7**. A discussion is presented on the methodologies and outcomes within this thesis, and recommendations are made for future research.

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